# OXYFLUORINATION

**THIS INVENTION** relates, broadly, to oxyfluorination. More particularly the invention relates to a process for the oxyfluorination of a surface of a solid to activate it.

According to the invention, there is provided a process for the activation by oxyfluorination of at least part of a surface of a solid, which process includes exposing, under selected conditions of temperature and pressure and for a selected reaction time, at least part of the surface of the material of the solid to an oxyfluorinating atmosphere which is a gas/vapour mixture which includes at least one fluorine-containing gas which reacts with the material of the exposed surface, at least one oxygen-containing gas which reacts with the material of the exposed surface, and water vapour, said gases in the oxyfluorinating atmosphere acting to oxyfluorinate the exposed surface, thereby to activate it, and the water vapour acting to enhance the activation.

By fluorine-containing gas is meant that each molecule of the gas contains at least one fluorine atom, and the term oxygen-containing gas has a corresponding meaning.

Instead, the process may include selecting the solid material which is subjected to activation by oxyfluorination from metals and metalloids which are members of the group consisting of mild steel, low carbon steel, stainless steel, and mixtures or alloys of any two or more thereof. In particular, the process may thus include selecting mild steel or low carbon steel as the solid material which is subjected to activation by oxyfluorination.

As will be appreciated and as indicated above, exposing the surface of the solid material to the oxyfluorinating atmosphere comprising the gas/vapour mixture of the present invention will be under conditions of temperature and pressure, and for a reaction time, selected to provide the exposed surface with desired properties such as, in particular, an enhanced amenability to adhesive bonding to other materials. In particular, the process may include selecting the fluorine-containing gas which reacts with the exposed surface from the group consisting of molecular fluorine ($F_2$), fluorinated noble gases, fluorohalogens, oxides of fluorine, and mixtures of any two or more thereof. More particularly, the process may include selecting the fluorine-containing gas from the group consisting of $F_2$, $XeF_2$, ClF, $ClF_3$, BrF, $BrF_3$, $BrF_5$, $IF_7$, $OF_2$, $O_2F_2$ and mixtures of any two or more thereof. In other words, the fluorine-containing gas may be molecular fluorine ($F_2$) itself, or it may be made up of one or more other suitable fluorine-containing gaseous compounds, examples of which are fluorinated noble gases such as $XeF_2$, or fluorohalogens such as ClF. $ClF_3$, BrF, $BrF_3$, $BrF_5$, and $IF_7$, or oxides of fluorine such as $OF_2$ or $O_2F_2$ so that, in other words, the

**CLAIMS:**

1. A process for the activation by oxyfluorination of at least part of a surface of a solid, which process includes exposing, under selected conditions of temperature and pressure and for a selected reaction time, at least part of the surface of the material of the solid to an oxyfluorinating atmosphere which is a gas/vapour mixture which includes at least one fluorine-containing gas which reacts with the material of the exposed surface, at least one oxygen-containing gas which reacts with the material of the exposed surface, and water vapour, said gases in the oxyfluorinating atmosphere acting to oxyfluorinate the exposed surface, thereby to activate it, and the water vapour acting to enhance the activation.

2. A process as claimed in Claim 1, in which the enhancement of the activation of the exposed surface acts to enhance the amenability of the exposed surface to adhesive bonding to other materials.

3. A process as claimed in Claim 1 or Claim 2, which includes selecting the solid material which is subjected to activation by fluorination from the group consisting of polymeric materials having constituents which are confined to carbon and hydrogen, elastomeric materials having constituents which are confined to carbon and hydrogen, polymeric materials having constituents which are not confined to carbon and hydrogen and which include, in addition to carbon and hydrogen, other atomic species as constituents, elastomeric materials having constituents which are not confined to carbon

and hydrogen and which include, in addition to carbon and hydrogen, other atomic species as constituents, carbon, glasses, metals, metalloids, wood, leather, cotton, wool, ceramics, asbestos and blends and mixtures thereof.

4. A process as claimed in Claim 3, which includes selecting the solid material which is subjected to activation by oxyfluorination from the group of materials consisting of polymeric materials, elastomeric materials and mixtures of any two or more thereof.

5. A process as claimed in Claim 4, which includes selecting the solid material which is subjected to activation by oxyfluorination from the group of materials having constituents which are confined to carbon and hydrogen.

6. A process as claimed in Claim 4, which includes selecting the solid material which is subjected to activation by oxyfluorination from the group of materials having constituents which are not confined to carbon and hydrogen and which include, in addition to carbon and hydrogen, other atomic species as constituents.

7. A process as claimed in Claim 3, which includes selecting the solid material which is subjected to activation by oxyfluorination from the group of materials consisting of carbon, glasses, metals, metalloids and mixtures of any two or more thereof.

8. A process as claimed in Claim 7, which includes selecting carbon as the material which is subjected to activation by oxyfluorination.

9. A process as claimed in Claim 7, which includes selecting the solid material which is subjected to activation by oxyfluorination from metals and metalloids which are members of the group consisting of mild steel, low carbon steel, stainless steel and mixtures or alloys of any two or more thereof.

10. A process as claimed in Claim 9, which includes selecting mild steel or low carbon steel as the solid material which is subjected to activation by oxyfluorination.

11. A process as claimed in any one of the preceding claims, which includes selecting the fluorine-containing gas which reacts with the exposed surface from the group consisting of molecular fluorine, fluorinated noble gases, fluorohalogens, oxides of fluorine, and mixtures of any two or more thereof.

12. A process as claimed in Claim 11, which includes selecting the fluorine-containing gas from the group consisting of $F_2$, $XeF_2$, ClF, $ClF_3$, BrF, $BrF_3$, $BrF_5$, $IF_7$, $OF_2$, $O_2F_2$ and mixtures of any two or more thereof.

13. A process as claimed in any one of the preceding claims, which includes selecting the oxygen-containing gas which reacts with the exposed surface from molecular oxygen, ozone and mixtures thereof.

14. A process as claimed in any one of the preceding claims, which includes diluting the oxyfluorinating atmosphere with a diluent gas which is inert to the exposed surface and inert to the other constituents of the oxyfluorinating atmosphere, and does not react therewith.

15. A process as claimed in Claim 14, which includes selecting the inert gas from the group consisting of nitrogen, the noble gases and mixtures of any two or more thereof.

16. A process as claimed in Claim 15, which includes selecting the inert gas from the group consisting of helium, argon, carbon dioxide, molecular nitrogen and mixtures of any two or more thereof.

17. A process as claimed in any one of the preceding claims, which includes using, as the oxyfluorinating atmosphere, a gas/vapour mixture of molecular fluorine, molecular oxygen and water vapour.

18. A process as claimed in Claim 17, which includes diluting the oxyfluorinating atmosphere, using molecular nitrogen as a diluent.

19. A process as claimed in any one of the preceding claims, which includes subjecting the oxyfluorinating atmosphere to ultra-violet radiation before the exposing of the solid material to the oxyfluorinating atmosphere is ended.

20. A process as claimed in Claim 19, in which the subjecting of the oxyfluorinating atmosphere to ultra-violet radiation is prior to the exposing of the solid material to the oxyfluorinating atmosphere.

21. A process as claimed in Claim 19 or Claim 20, in which the subjecting of the oxyfluorinating atmosphere to ultra-violet radiation is during the exposing of the solid material to the oxyfluorinating atmosphere.

22. A process as claimed in any one of the preceding claims, which includes exposing the solid material to a said oxyfluorinating atmosphere in which the fluorine-containing gas includes molecular fluorine at a partial pressure of 0.01 – 200 kPa.

23. A process as claimed in any one of the preceding claims, which includes exposing the solid material to the oxyfluorinating atmosphere for a period of 0.10 seconds – 10 hours, at a total pressure of the oxyfluorinating atmosphere of 0.1 – 500 kPa with the surface of the solid material and the oxyfluorinating atmosphere at a temperature at which the solid material has a surface which is stable.

24. A process as claimed in Claim 23, which includes exposing the solid material to the oxyfluorinating atmosphere at a total pressure of 1 – 200 kPa, and at a said temperature which is above 0°C, for a period of 0.1 seconds – 1 hour.

25. A process as claimed in Claim 24, which includes exposing the solid material to the oxyfluorinating atmosphere at a pressure 5 – 150 kPa and at a said temperature which is 20 – 100°C, for a period of 1 second – 10 minutes.

26. A process as claimed in any one of the preceding claims, which includes exposing the solid material to a said oxyfluorinating atmosphere which, in addition to its comprising a fluorine-containing gas, an oxygen-containing gas and water vapour, includes at least one further reactive constituent selected from the group consisting of halogens other than fluorine, interhalogen compounds and mixture of any two or more thereof.

27. A process as claimed in any one of the preceding claims, which includes exposing the solid material to a said oxyfluorinating atmosphere having a fluorine-containing gas content of 0.1 – 99% by volume.

28. A process as claimed in Claim 27, which includes exposing the solid material to a said oxyfluorinating atmosphere having a fluorine-containing gas content of 1 – 30% by volume.

29. A process as claimed in Claim 27 or Claim 28, which includes exposing the solid material to a said oxyfluorinating atmosphere in which the fluorine-containing gas forms 5 – 20% by volume and the oxygen-containing gas forms 5 – 95% by volume.

30. A process as claimed in any one of Claim 27 – 29 inclusive, which includes exposing the solid material to a said oxyfluorinating atmosphere which has a relative humidity of 0.1 – 99%.

31. A process as claimed in Claim 30, which includes exposing the solid material to a said oxyfluorinating atmosphere which has a relative humidity of 30-90%.

32. A process as claimed in Claim 30, which includes exposing the solid material to a said oxyfluorinating atmosphere which has a relative humidity of 50 – 80%.

33. A process as claimed in any one of the preceding claims, which includes exposing the solid material to the oxyfluorinating atmosphere until the surface concentration of fluorine of said exposed surface has been increased by at least 0.01 $\mu gF/cm^2$.

34. A process as claimed in Claim 33, which includes exposing the solid material to the oxyfluorinating atmosphere until the surface concentration of fluorine of said exposed surface has been increased by 0.01 – 50 $\mu gF/cm^2$.

35. A process as claimed in any one of the preceding claims, which includes, prior to the exposing of the solid material to the oxyfluorinating atmosphere, degreasing the exposed surface.

36. A process as claimed in any one of the preceding claims, which includes exposing the solid material to the oxyfluorinating atmosphere in a reaction chamber in a reaction vessel, and which includes flushing the reaction chamber by means of the oxyfluorinating atmosphere prior to the exposing of the solid material to the oxyfluorinating atmosphere.

37. A process as claimed in Claim 1, substantially as described herein.

38. An oxyfluorinated product whenever produced by the process of any one of Claims 1 – 37 inclusive.

39. A product as claimed in Claim 38, substantially as described herein.

# INTERNATIONAL SEARCH REPORT

PCT/IB 03/04701

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C08J7/06 C08J7/12

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C08J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category * | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
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| X | US 2001/009176 A1 (MORI YOSHIAKI ET AL)<br>26 July 2001 (2001-07-26)<br>paragraph '0142! - paragraph '0143! | 1-39 |
| X | PATENT ABSTRACTS OF JAPAN<br>vol. 008, no. 043 (M-279),<br>24 February 1984 (1984-02-24)<br>& JP 58 199132 A (NIPPON MEKTRON KK),<br>19 November 1983 (1983-11-19)<br>abstract | 1-39 |
| X | GB 1 488 931 A (AIR PROD & CHEM)<br>19 October 1977 (1977-10-19)<br>page 6, line 33 - line 38 | 1-39 |
| X | US 4 800 053 A (MARSH PAUL D ET AL)<br>24 January 1989 (1989-01-24)<br>example 1 | 1-39 |

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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| Date of the actual completion of the international search | Date of mailing of the international search report |
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